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(54) Title: HIGH HOMOGENEITY SILICA GLASS PREPARED THROUGH A SOL-GEL PROCEDURE

(57) Abstract: The present invention relates to a Si₂O glass characterized by a high homogeneity, prepared through a sol-gel procedure.

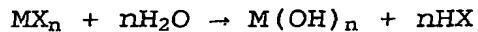
HIGH HOMOGENEITY SILICA GLASS PREPARED THROUGH A SOL-GEL PROCEDURE

5 The present invention relates to a highly homogeneous Si₂O glass prepared through a sol-gel procedure.

The sol-gel term defines a wide variety of processes which, even if being different as far as the working details or the reagents are concerned, are characterized by the following common operations:

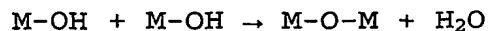
10 - preparation of a solution, or a suspension, of a precursor formed by a compound of the element (M) the oxide of which has to constitute the final glassy article;

15 - hydrolysis, acid or base catalyzed, of the precursor, inside the solution or suspension, to form M-OH groups according to the reaction



20 wherein X generally is an alcohol residue and n means the element M valence; the alcoxydes M(OR)_n can be replaced by soluble salts of the element M such as chlorides or nitrates, and, in some cases, oxydes. The obtained mixture, i.e. a solution or a colloidal suspension, is named sol;

25 - polycondensation of the M-OH groups according to the reaction



which requires a time from few seconds to some days, depending on the solution composition and the temperature; during this step, a matrix is formed

called, case by case, alcohogel, hydrogel or more generally, gel;

- gel drying till the formation of a porous monolithic body; during this step, the solvent is removed through a simple controlled evaporation, which determines the so called xerogel, or through an extraction in autoclave which determines the so called aerogel; the obtained body is a porous glass, which may have an apparent density of 10% to about 50% of the theoric density of the oxide having the same composition; the dried gel can be industrially used as such;
- densification of the dried gel by a treatment at a temperature, generally ranging between 800°C and 1500°C, depending on the gel chemical composition and the preceding step process parameters; during this step the porous gel is becoming dense, under a controlled atmosphere, till to obtain a glassy or ceramic compact oxide having the theoric density, with a linear shrinkage equal to about 50%.

20 The final densification let a glassy product be obtained having good general characteristics, and, however, without any such optical homogeneity property to let the material be crossed by the transmitted light wave front without any suffered distortion.

25 The Applicant has found that in the case suitable treatments under controlled atmosphere are carried out during the densification stage, the final glassy product is obtained having no streak and strip, the same being consequently characterized by an almost total homogeneity.

Therefore, the object of the present invention is a silica glass characterized, inter alia, by the following specific properties:

- light internal transmittance in the wave length between 185nm and 193nm higher than 85%
- 5 - light internal transmittance in the wave length between 193nm and 2600nm higher than 99.5%
- light internal transmittance in the wave length between 2600nm and 2730nm higher than 99%
- 10 - light internal transmittance in the wave length between 2730nm and 3200nm higher than 85%
- no streak, material of class 4 or better according to the rule DIN ISO 10110-4
- no strip
- 15 - no signal in the shadography (no shadow or intensity change)

such a silica glass being prepared according to a sol-gel process wherein, in the meanwhile the densification is achieved, a treatment is carried out by means of an 20 atmosphere containing water traces.

Claims

1. Silica glass characterized by the following specific properties:

5 - light internal transmittance in the wave length between 185nm and 193nm higher than 85%

- light internal transmittance in the wave length between 193nm and 2600nm higher than 99.5%

- light internal transmittance in the wave length 10 between 2600nm and 2730nm higher than 99%

- light internal transmittance in the wave length between 2730nm and 3200nm higher than 85%

- no streak, material of class 4 or better according to the rule DIN ISO 10110-4

15 - no strip

- no signal in the shadography (no shadow or intensity change)

such a silica glass being prepared according to a sol-gel process wherein, in the meanwhile the densification 20 is achieved, a treatment is carried out by means of an atmosphere containing water traces.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C03C1/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 063 179 A (MENASHI JAMEEL ET AL) 5 November 1991 (1991-11-05) the whole document -----	1
X	TSENG T Y ET AL: "Various atmosphere effects on sintering of compacts of SiO ₂ /sub 2/ microspheres" J. MATER. SCI. (UK), JOURNAL OF MATERIALS SCIENCE, OCT. 1986, UK, vol. 21, no. 10, 1986, pages 3615-3624, XP001189680 ISSN: 0022-2461 the whole document -----	1
X	US 5 068 208 A (HAUN NIELS ET AL) 26 November 1991 (1991-11-26) column 6, line 6 - line 14 ----- -/-	1

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Patent family members are listed in annex.

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INTERNATIONAL SEARCH REPORTInternational Application No
PCT/EP2004/002577**C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT**

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 310 486 A (SHINETSU CHEMICAL CO) 5 April 1989 (1989-04-05) column 8, line 37 - column 9, line 4 -----	1
X	US 4 681 615 A (KANBE SADAO ET AL) 21 July 1987 (1987-07-21) examples -----	1
X	US 6 360 564 B1 (UKRAINCYK LJERKA ET AL) 26 March 2002 (2002-03-26) column 7, line 45 - line 50 -----	1

INTERNATIONAL SEARCH REPORT

Information on patent family members

 International Application No
 PCT/EP2004/002577

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
US 5063179	A	05-11-1991	AT	115519 T	15-12-1994
			AU	7481291 A	18-09-1991
			DE	69105955 D1	26-01-1995
			DE	69105955 T2	20-04-1995
			EP	0517841 A1	16-12-1992
			JP	5505167 T	05-08-1993
			JP	3274466 B2	15-04-2002
			WO	9113040 A1	05-09-1991
US 5068208	A	26-11-1991	NONE		
EP 0310486	A	05-04-1989	JP	1087522 A	31-03-1989
			JP	7035256 B	19-04-1995
			JP	1087523 A	31-03-1989
			JP	2004371 C	20-12-1995
			JP	7035257 B	19-04-1995
			DE	3869308 D1	23-04-1992
			EP	0310486 A2	05-04-1989
			US	4883521 A	28-11-1989
US 4681615	A	21-07-1987	JP	1024735 B	12-05-1989
			JP	1542618 C	15-02-1990
			JP	60065732 A	15-04-1985
			JP	60065733 A	15-04-1985
			JP	1573634 C	20-08-1990
			JP	59116135 A	04-07-1984
			JP	64000331 B	06-01-1989
			JP	1833144 C	29-03-1994
			JP	60131833 A	13-07-1985
			DE	3390375 C2	30-11-1989
			DE	3390375 T	07-02-1985
			EP	0131057 A1	16-01-1985
			GB	2140408 A , B	28-11-1984
			HK	69589 A	08-09-1989
			WO	8402519 A1	05-07-1984
			NL	8320410 A , C	01-11-1984
			NL	8320410 T	01-11-1984
			SG	39588 G	27-01-1989
			US	4801318 A	31-01-1989
US 6360564	B1	26-03-2002	AU	1919101 A	31-07-2001
			CA	2396955 A1	26-07-2001
			CN	1423623 T	11-06-2003
			EP	1254078 A1	06-11-2002
			JP	2003520180 T	02-07-2003
			WO	0153223 A1	26-07-2001